



# Analytical Laboratory

Page 1 of 17

13339 Hagers Ferry Road  
Huntersville, NC 28078-7929  
McGuire Nuclear Complex - MG03A2  
Phone: 980-875-5245 Fax: 980-875-4349

## Order Summary Report

**Order Number:** J13110185

Project Name: WWTS FGD-Routine 2013

Customer Name(s): Bill K, Wayne C, and Melonie M

Customer Address: 3195 Pine Hall Rd  
Mailcode: Belews Steam Station  
Belews Creek, NC 28012

Lab Contact: Jason C Perkins Phone: 980-875-5348

**Report Authorized By:** \_\_\_\_\_ **Date:** 12/5/2013  
(Signature) Jason C Perkins

---

### Program Comments:

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

### Data Flags & Calculations:

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted. Subcontracted data included on the Duke Certificate of Analysis is to be used as information only. Certified vendor results can be found in the subcontracted lab final report. Duke Energy Analytical Laboratory subcontracts analyses to other vendor laboratories that have been qualified by Duke Energy to perform these analyses except where noted.

### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

*Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)*

### Certification:

The Analytical Laboratory holds the following State Certifications : North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

Sample ID's & Descriptions:

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2013027567	BELEWS	08-Nov-13 9:40 AM	Matt C.	FGD Purge Eff
2013027568	BELEWS	08-Nov-13 9:46 AM	Matt C.	EQ Tank Eff
2013027569	BELEWS	08-Nov-13 9:50 AM	Matt C.	BioReactor 1 Inf
2013027570	BELEWS	08-Nov-13 9:55 AM	Matt C.	BioReactor 2 Inf
2013027571	BELEWS	08-Nov-13 10:00 AM	Matt C.	BioReactor 2 Eff
2013027572	BELEWS	08-Nov-13 10:30 AM	Matt C.	Filter Blk
2013027573	BELEWS	31-Oct-13 2:10 PM	D. Baker	TRIP BLANK
7 Total Samples				

## Technical Validation Review

### Checklist:

- |  |   |  |
|--|---|--|
| COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures). | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No            |
| All Results are less than the laboratory reporting limits.   | <input type="checkbox"/> Yes            | <input checked="" type="checkbox"/> No |
| All laboratory QA/QC requirements are acceptable.  | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No            |

### Report Sections Included:

- |   |  |
|---|--|
| <input checked="" type="checkbox"/> Job Summary Report                            | <input checked="" type="checkbox"/> Sub-contracted Laboratory Results                |
| <input checked="" type="checkbox"/> Sample Identification                         | <input type="checkbox"/> Customer Specific Data Sheets, Reports, & Documentation     |
| <input checked="" type="checkbox"/> Technical Validation of Data Package          | <input type="checkbox"/> Customer Database Entries                                   |
| <input checked="" type="checkbox"/> Analytical Laboratory Certificate of Analysis | <input checked="" type="checkbox"/> Chain of Custody                                 |
| <input type="checkbox"/> Analytical Laboratory QC Report                          | <input checked="" type="checkbox"/> Electronic Data Deliverable (EDD) Sent Separatel |

Reviewed By: DBA Account

Date: 12/5/2013

# Certificate of Laboratory Analysis

Page 4 of 17

*This report shall not be reproduced, except in full.***Order # J13110185**

Site: FGD Purge Eff

Collection Date: 08-Nov-13 9:40 AM

**Sample #: 2013027567**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>NITRITE + NITRATE (COLORIMETRIC)</u></b>								
Nitrite + Nitrate (Colorimetric)	2.4	mg-N/L		0.05	5	EPA 353.2	11/15/2013 11:52	BGN9034
<b><u>INORGANIC IONS BY IC</u></b>								
Bromide	230	mg/L		5	50	EPA 300.0	11/22/2013 16:24	JAHERMA
<b><u>MERCURY (COLD VAPOR) IN WATER</u></b>								
Mercury (Hg)	263	ug/L		5	100	EPA 245.1	11/23/2013 10:29	DKJOHN2
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	207	mg/L		0.5	10	EPA 200.7	11/14/2013 09:31	MHH7131
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	935	ug/L		10	10	EPA 200.8	11/20/2013 14:16	DJSULL1
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	234	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1
Chromium (Cr)	442	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1
Copper (Cu)	166	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1
Nickel (Ni)	277	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1
Selenium (Se)	4970	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1
Zinc (Zn)	316	ug/L		10	10	EPA 200.8	11/20/2013 11:45	DJSULL1

**SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)**

Vendor Parameter	Complete	Vendor Method	V_AS&C
------------------	----------	---------------	--------

Site: EQ Tank Eff

Collection Date: 08-Nov-13 9:46 AM

**Sample #: 2013027568**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>MERCURY (COLD VAPOR) IN WATER</u></b>								
Mercury (Hg)	133	ug/L		2.5	50	EPA 245.1	11/23/2013 10:32	DKJOHN2
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	200	mg/L		0.5	10	EPA 200.7	11/14/2013 09:35	MHH7131
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	540	ug/L		10	10	EPA 200.8	11/20/2013 14:19	DJSULL1

# Certificate of Laboratory Analysis

Page 5 of 17

*This report shall not be reproduced, except in full.***Order # J13110185**

Site: EQ Tank Eff

Collection Date: 08-Nov-13 9:46 AM

**Sample #: 2013027568**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b>TOTAL RECOVERABLE METALS BY ICP-MS</b>								
Arsenic (As)	123	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1
Chromium (Cr)	237	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1
Copper (Cu)	88.9	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1
Nickel (Ni)	189	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1
Selenium (Se)	2650	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1
Zinc (Zn)	175	ug/L		10	10	EPA 200.8	11/20/2013 11:49	DJSULL1

Site: BioReactor 1 Inf

Collection Date: 08-Nov-13 9:50 AM

**Sample #: 2013027569**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>NITRITE + NITRATE (COLORIMETRIC)</u></b>								
Nitrite + Nitrate (Colorimetric)	2.7	mg-N/L		0.05	5	EPA 353.2	11/15/2013 11:53	BGN9034
<b><u>Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	186	mg/L		0.5	10	EPA 200.7	11/14/2013 09:40	MHH7131
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	122	ug/L		10	10	EPA 200.8	11/20/2013 14:23	DJSULL1
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
Nickel (Ni)	16.5	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
Selenium (Se)	117	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	11/20/2013 11:52	DJSULL1
<b><u>SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	Complete					Vendor Method		V_AS&C

# Certificate of Laboratory Analysis

Page 6 of 17

*This report shall not be reproduced, except in full.***Order # J13110185**

Site: BioReactor 2 Inf

Collection Date: 08-Nov-13 9:55 AM

**Sample #: 2013027570**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	<b>Complete</b>	ug/l				Vendor Method		V_AS&C
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	<b>198</b>	mg/L		0.5	10	EPA 200.7	11/14/2013 09:44	MHH7131
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1
Cadmium (Cd)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1
Chromium (Cr)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1
Copper (Cu)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1
Nickel (Ni)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1
Selenium (Se)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1
Silver (Ag)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1
Zinc (Zn)	<b>&lt; 10</b>	ug/L		10	10	EPA 200.8	11/20/2013 11:56	DJSULL1

Site: BioReactor 2 Eff

Collection Date: 08-Nov-13 10:00 AM

**Sample #: 2013027571**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>NITRITE + NITRATE (COLORIMETRIC)</u></b>								
Nitrite + Nitrate (Colorimetric)	<b>&lt; 0.01</b>	mg-N/L		0.01	1	EPA 353.2	11/15/2013 11:41	BGN9034
<b><u>INORGANIC IONS BY IC</u></b>								
Bromide	<b>220</b>	mg/L		5	50	EPA 300.0	11/22/2013 16:43	JAHERMA
<b><u>Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	<b>Complete</b>	ug/l				Vendor Method		V_AS&C
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	<b>194</b>	mg/L		0.5	10	EPA 200.7	11/14/2013 09:48	MHH7131
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1
Cadmium (Cd)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1
Chromium (Cr)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1
Copper (Cu)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1
Nickel (Ni)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1
Selenium (Se)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1
Silver (Ag)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1
Zinc (Zn)	<b>&lt; 5</b>	ug/L		5	5	EPA 200.8	11/20/2013 11:59	DJSULL1

# Certificate of Laboratory Analysis

Page 7 of 17

*This report shall not be reproduced, except in full.***Order # J13110185**

Site: BioReactor 2 Eff

Collection Date: 08-Nov-13 10:00 AM

**Sample #: 2013027571**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	Complete					Vendor Method		V_AS&C
<b><u>TOTAL DISSOLVED SOLIDS</u></b>								
TDS	14000	mg/L		25	1	SM2540C	11/19/2013 14:19	DSBAKE1

Site: Filter Blk

Collection Date: 08-Nov-13 10:30 AM

**Sample #: 2013027572**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 13:38	DJSULL1

Site: TRIP BLANK

Collection Date: 31-Oct-13 2:10 PM

**Sample #: 2013027573**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	< 0.05	mg/L		0.05	1	EPA 200.7	11/14/2013 09:57	MHH7131
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1
Cadmium (Cd)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1
Chromium (Cr)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1
Copper (Cu)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1
Nickel (Ni)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1
Selenium (Se)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1
Silver (Ag)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1
Zinc (Zn)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 11:39	DJSULL1



**APPLIED SPECIATION  
AND CONSULTING, LLC**

18804 Northcreek Parkway Bothell, WA, 98011  
Tel: (425) 483-3300 Fax: (425) 483-9818  
[www.appliedspeciation.com](http://www.appliedspeciation.com)

December 3, 2013

Jay Perkins  
Duke Energy Analytical Laboratory  
Mail Code MGO3A2 (Building 7405)  
13339 Hagers Ferry Rd.  
Huntersville, NC 28078  
(704) 875-5245

Project: Belews - FGD WWTS (Bi-Monthly & Flex Fuel & DSI) (LIMS# J13110185)

Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for total mercury and selenium speciation analysis on November 13, 2013. The samples were received in a sealed cooler at 0.1°C on November 14, 2013. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). Mercury quantitation was performed via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

A handwritten signature in black ink, appearing to read "Jeremy Maute".

Jeremy Maute  
Project Coordinator  
Applied Speciation and Consulting, LLC



Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins  
Duke Energy Analytical Laboratory  
Mail Code MGO3A2 (Building 7405)  
13339 Hagers Ferry Rd.  
Huntersville, NC 28078

Project: Belews - FGD WWTS (Bi-Monthly & Flex Fuel & DSI) (LIMS# J13110185)

December 3, 2013

## 1. Sample Reception

Four (4) aqueous samples were submitted for selenium speciation analysis on November 13, 2013. Three (3) additional samples were submitted for total mercury quantitation. All samples were received in acceptable condition on November 14, 2013 in a sealed container at 0.1°C.

All samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and were designated discrete sample identifiers. The 40mL borosilicate glass vials submitted for total mercury were preserved with bromine monochloride (BrCl) solution. The resulting samples were stored in a secure polyethylene container, known to be free from trace metals contamination, until the analyses could be performed.

An aliquot of each sample requiring selenium speciation evaluation was filtered (0.45µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS).

Two 40 mL glass vials were received that listed a LIMS ID of 2013027570. Both bottles were labeled with a sample ID of BioReactor 2 Inf and both bottles listed a collection date/time of (11/8/13, 0955). These descriptive parameters match the BioReactor 2 Inf sample entry on the chain-of-custody (COC) form associated with LIMS ID J13110185. There was no way to discern between the two samples visibly. The COC form associated with LIMS ID J13110187 was also present in the cooler and the COC indicated that the client sample (requesting total mercury analysis) identified as (2013027578, BioReactor 2 Inf, 11/11/13, 0745) should be present. A 40 mL glass container with the client sample BioReactor 2 Inf (LIMS ID 2013027578) was absent from the cooler. The two identically labeled samples requesting total mercury analysis were labeled internally by the laboratory sample reception staff at Applied Speciation as (2013027570-BioReactor 2 Inf A) and

(2013027570-BioReactor 2 Inf B). The samples were analyzed for total mercury yielding similar values, 0.0363 µg/L and 0.0296 µg/L, respectively.

The client was informed of this sample ID issue. Since the total mercury results were similar in quantity, Applied Speciation was directed to report the total mercury result for (2013027570-BioReactor 2 Inf A) in the report associated with LIMS ID J13110185. Applied Speciation was instructed to report the total mercury result for (2013027570-BioReactor 2 Inf B) in the report associated with LIMS ID J13110187.

## 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

Total Mercury Quantitation by CV-ICP-MS All samples and preparation blanks for total mercury quantitation were preserved with 2% (v/v) BrCl. The resulting samples were analyzed for mercury via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS).

Selenium Speciation Analysis by IC-ICP-CRC-MS Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

## 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimum interval of every ten analytical runs.

Total Mercury Quantitation by CV-ICP-MS The sample fractions for total mercury quantitation were analyzed by cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS) on November 18, 2013. Aliquots of each sample are reacted with a reductant in-line and transported to a gas-liquid separator. The volatile elemental mercury that is formed is then swept by a stream of argon gas into a radio frequency (RF) plasma where

energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and separated on the basis of their mass-to-charge ratio ( $m/z$ ) by a mass spectrometer. A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

*Selenium Speciation Analysis by IC-ICP-CRC-MS* Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS) on November 19, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic ( $\text{pH} > 7$ ) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (CRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios ( $m/z$ ). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

#### **4. Analytical Issues**

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

The eMDL for mercury has been calculated using the standard deviation of the preparation blanks preserved and analyzed concurrently with the submitted samples.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

A handwritten signature in black ink, appearing to read 'J. Maute', with a stylized flourish extending to the right.

Jeremy Maute  
Project Coordinator  
Applied Speciation and Consulting, LLC

Total Mercury & Selenium Speciation Results for Duke Energy  
 Project Name: Belews - FGD WWTS (Bi-Monthly & Flex Fuel & DSI)  
 Contact: Jay Perkins  
 LIMS #J13110185

Date: December 3, 2013  
 Report Generated by: Jeremy Maute  
 Applied Speciation and Consulting, LLC

**Sample Results**

Sample ID	Total Hg	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Unknown Se Species (n)
FGD Purge Eff	NR	1000	24.9	7.5	2.8	ND (< 1.7)	4.3 (1)
BioReactor 1 Inf	0.129	56.6	32.9	ND (< 0.53)	2.32	ND (< 0.43)	0.71 (1)
BioReactor 2 Inf	0.0363	NR	NR	NR	NR	NR	NR
BioReactor 2 Eff	0.0058	ND (< 0.47)	ND (< 0.30)	ND (< 0.53)	ND (< 0.43)	ND (< 0.43)	0 (0)

All results reflect the applied dilution and are reported in µg/L

NR = Analysis not requested

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

Total Mercury & Selenium Speciation Results for Duke Energy  
 Project Name: Belews - FGD WWTS (Bi-Monthly & Flex Fuel & DSI)  
 Contact: Jay Perkins  
 LIMS #J13110185

Date: December 3, 2013  
 Report Generated by: Jeremy Maute  
 Applied Speciation and Consulting, LLC

**Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 5x	eMDL 250x	eMDL 1000x
Hg	-0.0003	-0.0002	-0.0007	-0.0004	-0.0004	0.0002	0.0001	0.0006	-	-
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.47	1.9
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.001	-	0.30	1.2
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.53	2.1
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.43	1.7
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.43	1.7

eMDL = Estimated Method Detection Limit

\*Please see narrative regarding eMDL calculations

**Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Hg	NIST 1641d	1568	1626	103.7
Se(IV)	LCS	9.57	9.28	96.9
Se(VI)	LCS	9.48	9.22	97.3
SeCN	LCS	8.92	8.55	95.9
MeSe(IV)	LCS	6.47	6.23	96.3
SeMe	LCS	9.32	8.84	94.8

Total Mercury & Selenium Speciation Results for Duke Energy  
 Project Name: Belews - FGD WWTS (Bi-Monthly & Flex Fuel & DSI)  
 Contact: Jay Perkins  
 LIMS #J13110185

Date: December 3, 2013  
 Report Generated by: Jeremy Maute  
 Applied Speciation and Consulting, LLC

**Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Hg	Batch QC	0.1934	0.1955	0.1945	1.1
Se(IV)	BioReactor 2 Eff	ND (< 0.47)	ND (< 0.47)	NC	NC
Se(VI)	BioReactor 2 Eff	ND (< 0.30)	ND (< 0.30)	NC	NC
SeCN	BioReactor 2 Eff	ND (< 0.53)	ND (< 0.53)	NC	NC
MeSe(IV)	BioReactor 2 Eff	ND (< 0.43)	ND (< 0.43)	NC	NC
SeMe	BioReactor 2 Eff	ND (< 0.43)	ND (< 0.43)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

**Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Hg	Batch QC	2.000	2.347	107.6	2.000	2.320	106.3	1.2
Se(IV)	BioReactor 2 Eff	1390	1336	96.1	1390	1344	96.7	0.6
Se(VI)	BioReactor 2 Eff	1261	1176	93.3	1261	1190	94.3	1.1
SeCN	BioReactor 2 Eff	1144	1026	89.7	1144	1043	91.2	1.7



pg 10/3

**Duke Energy Analytical Laboratory**  
Mail Code MGO3A2 (Building 7405)  
13339 Hagers Ferry Rd  
Huntersville, N. C. 28078  
(704) 875-5245  
Fax: (704) 875-4349

<b>AS&amp;C</b> <b>PO #650910</b>	Cooler Temp. (C) 1=Preserv. 1=HCl 2=H <sub>2</sub> SO <sub>4</sub> 3=HNO <sub>3</sub> 4=lbc 5=None	4	4 3,4 3,4 2,4	4	15 Analyses Required	9 245.1**	filtered	-AS&C)	UST _____ RCRA Waste _____

Se Speciation Bottle ID	<sup>13</sup> Sample Description or ID
	FGD Purge Eff
	EQ Tank Eff.
	BioReactor 1 Inf
	BioReactor 2 Inf
	BioReactor 2 Eff
	Filter Blk
	Metals Trip Blk

Sampling conducted: 2nd and 4th Vestiges day	Date	Time	Signature
	11/8/13	0940	MC
		0946	MC
		0950	MC
		0955	MC
		1000	MC
	11/8/13	1030	MC
	10/12/13	1410	D. Baker

Filtering







<sup>17</sup> Comp.	<sup>18</sup> Grab	TDS	Br (Dione)	Metals* +
			1	1
				1
				1**
				1**
		1	1	1**
				1**

Se (IMS)	NO3-NO2	Hg 200.8	Se, spec	AsAC (Impc)	bolle back
1	1		1		
1	1	1	1		
1	1	1	1		
	1	1	1		
1					

did please provide a filter blank too.

Team Kit to Travis Thornton © Deleva

Customer to sign & date below - Fill out from left to right.

1) Requiring By 16.1 Cass-CT	Date/Time 11-11-2013 15:00	2) Accepted By 	Date/Time 4/24/13	320
3) Requiring By 	Date/Time 4/3/13	4) Accepted By 	Date/Time	
5) Requiring By 	Date/Time 13/5	6) Accepted By	Date/Time	
7) Requiring By	Date/Time	8) Accepted By	Date/Time	
9) Seal Locked By 	Date/Time 4/13/13	10) Seal/Lock Opened By 	Date/Time 11/14/13	0.1°C
11) Seal Locked By	Date/Time	12) Seal/Lock Opened By	Date/Time	
Comments				

\* B by TRM/ICP As, Cd, Cr, Cu, Ni, Se, Ag, Zn by TRM/IMS 1<sup>st</sup>=No Hg

\* B by TRM/ICP As, Cd, Cr, Cu, Ni, Se, Ag, Zn by TRM/IMS <sup>1</sup> = No Hg

① Duplicate Hq sample Present

**22 Requested Turnaround**

21 Days

7 Days

- 48 Hr -

Other 14 day

1.

Page 16 of 17

21/5e/11



# CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM



## Duke Energy Analytical Laboratory

Mail Code MGO3A2 (Building 7405)  
13339 Hagers Ferry Rd  
Huntersville, N. C. 28078  
(704) 875-5245  
Fax: (704) 875-4349

## Analytical Laboratory Use Only

ORDER# 313110185	MATRIX: OTHER	Samples Originating From	NC SC
Logged By Dm	Date & Time 11/12/13 1315	SAMPLE PROGRAM Water	Ground NPDES
AS&C PO #650910		Drinking Water UST	RCRA Waste
Cooler Temp (C) 08		Preserv.: 1=HCL 2=H <sub>2</sub> SO <sub>4</sub> 3=HNO <sub>3</sub> 4=Ice 5=None	

Page 1 of 2 Page 17 of 17

**DISTRIBUTION**  
ORIGINAL to LAB,  
COPY to CLIENT

1) Project Name <b>Belews - FGD</b> <b>WWTS (Bi-Monthly &amp; Flex Fuel &amp; DSI)</b>	2) Phone No:
2) Client: <b>Bill Kennedy, Melonie Martin, Wayne Chapman</b>	Use Project: <b>WWTS FGD-Routine 2013</b>
5) Business Unit: 20003	6) Process: BMCEFGD
8) Oper. Unit: BC00	10) Reso. Center:

Customer to complete all  
appropriate non-shaded areas.

Sampling conducted: 2nd and 4th Wednesday

LAB USE ONLY
11) Lab ID
2013027567A
2013027568
2013027569
2013027570
2013027571
2013027572
2013027573

Se Speciation Bottle ID	13 Sample Description or ID	Date	Time	Signature	17 Comp.	18 Grab	TDS	Br (Dionex)	Metals* + Hg 245.1**	Se (IMS), filtered	NO3-NO2	Hg 200.8 (V_AS&C)	Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies)
	FGD Purge Eff	11/8/13	0940	MC				1	1	1	1		1
	EQ Tank Eff.		0946	MC					1	1			
	BioReactor 1 Inf		0950	MC					1**	1	1	1	1
	BioReactor 2 Inf		0955	MC					1**			1	
	BioReactor 2 Eff		1000	MC			1	1	1**		1	1	1
	Filter Blk	11/8/13	1030	MC						1			
	Metals Trip Blk	10/11/13	1410	D. Baker					1**				

Filtering of the Se is performed in the field please provide a filter blank too.

Return Kit to Travis Thorton @ Belews

1) Relinquished By Shil Goss-CJT	Date/Time 11-11-2013 15:00	2) Accepted By [Signature]	Date/Time 11/26/13 1300
3) Relinquished By [Signature]	Date/Time 11/13/13 1315	4) Accepted By [Signature]	Date/Time
5) Relinquished By	Date/Time	6) Accepted By	Date/Time
7) Relinquished By	Date/Time	8) Accepted By	Date/Time
9) Seal/Locked By	Date/Time	10) Seal/Lock Opened By	Date/Time
11) Seal/Locked By	Date/Time	12) Seal/Lock Opened By	Date/Time
Comments * B by TRM/ICP As, Cd, Cr, Cu, Ni, Se, Ag, Zn by TRM/IMS 1**=No Hg			

Customer, IMPORTANT!  
Please indicate desired turnaround.

## 22 Requested Turnaround

21 Days \_\_\_\_\_

\*7 Days \_\_\_\_\_

- 48 Hr \_\_\_\_\_

\*Other \_\_\_\_\_

\* Add. Cost Will Apply